NASA TECH BRIEF

Combining Micro Dry Column Chromatography and Mass Spectrometry

The problem:

To provide a rapid means for separating and identifying the colorless constituents of soluble mixtures. Sample preparation techniques should be fast and amenable to field use.

The solution:

A microscale technique which minimizes the time required for both preparation and analysis and is compatible with quantitative instruments such as the mass spectrometer. These techniques provide a means for fast analysis of soluble mixtures based upon the rapid fabrication of microcolumns in batches from readily available, storable materials. The analytic procedure becomes noncritical because of the short time required in repeating the steps. An important feature contributing to speed is the small size of the columns (e.g., 45 to 50 mm long, 0.7 mm i.d.). Capillary attraction and thus separation along the length of the column are therefore possible in a very short time (3 to 15 min. depending on the solvent system used). This is much faster than thin layer chromatography (TLC) which takes 30 minutes to 4 hours, and is more convenient to use and store than TLC. The extensive data relating to separation by TLC is directly applicable to the microcolumn technique.

How it's done:

Application of the principles of dry column chromatography in microscale has produced techniques for preparing microcolumns from glass pipettes filled with finely sieved adsorbents. Techniques include developing each column by inserting the wick into a solvent which carries the sample up through the column by capillary action. The columns are scribed at the separation points, and immediately after development are broken into equal sections which are subsequently introduced into the mass spectrometer via a direct insertion inlet system. Normally, samples are analyzed at a relatively low resolution of $M/\Delta M \cong 2.000$ which allows only limited mass measurement. This, however, is sufficient for identification of the majority of the bands. If required, the sample can be rerun, since only a very small amount of sample and time are consumed. The analyzer can thus cut out a specific, still unidentified band and investigate it under conditions of high resolving power $M/\Delta M > 10{,}000$ and make an identification after precise mass measurement of all peaks of the spectrum.

The mass spectrometer employed in these experiments is a double focusing instrument fitted with a direct insertion probe for introduction of solid samples. The probe is modified to handle the microcolumn sections directly and provide independent temperature control of the sample. Limits of detection and identification are $0.01\mu g$ or less at a resolving power of less than 2000 and 0.1 to $1.0\mu g$ at a resolving power of 10,000 to 20,000. The simple presence of a band can be detected below the 10 nanogram level.

Columns are broken into the mass spectrograph solid sample probe cup, using a counterbored adapter which slips over the top of the cup. Cups are then stored in the dark under an inert atmosphere at low temperature in large numbers until needed for analysis.

Notes:

1. In addition to being compatible with the mass spectrophotometer, this method can easily be adapted to gas chromatographic techniques.

(continued overleaf)

2. No additional documentation is available. Specific questions, however, may be directed to:
   Technology Utilization Officer
   NASA Pasadena Office
   4800 Oak Grove Drive
   Pasadena, California 91103
   Reference: B70-10231

Patent status:

Inquiries about obtaining rights for the commercial use of this invention may be made to NASA, Code GP, Washington, D.C. 20546.

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